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This report results from a contract tasking University of Glasgow as follows: The contractor will investigate quantum cascade lasers fabricated using a new native oxide technique called planar selective wet oxide fabrication. This technique will be employed to fabricate arrays of such lasers on a single chip to obtain high powered laser bars operating at wavelengths around five microns.

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1 Background and objectives

The quantum cascade laser (QCL) is an electrically pumped semiconductor laser that emits in the mid-IR part of the spectrum. Unlike most semiconductor lasers, which use electron-hole recombination to emit light, the QCL is unipolar and light emission takes place when electrons undergo transitions between energy levels that are all in the conduction band[†]. The QCL was invented and first fabricated by Jérôme Faist *et al.* at Bell Laboratories[1], based on a 1971 design for an electrically pumped intersubband optical amplifier[2]. Currently, QCLs have been made to lase at wavelengths ranging from ~3.5 μm to ~24 μm.

The wet thermal native oxides of Al containing III-V semiconductors have seen much use in photonic semiconductor devices, as they are electrically insulating and their low refractive indices (typically ~1.6) relative to III-V semiconductors (typically ~3) make them useful for forming optical waveguides. Wet thermal oxidation is the reaction of the semiconductor with steam, usually in an inert carrier gas such as N_2, and was developed for silicon in 1955. For III-V semiconductors, most work has been done with AlGaAs. Whilst this material has be used to fabricate QC lasers, better performance is obtained using the lattice-matched InP/InGaAs/InAlAs system. The wet thermal oxidation of InAlAs was first carried out by Caracci *et al.*[3] and has been used to fabricate QC lasers by Farmer *et al.*[4] With the wet thermal oxidation process, it is possible to oxidise from the surface the semiconductor down towards the substrate or to laterally oxidise a buried layer from an exposed edge. The former process is sometimes termed planar selective wet oxidation (PSWOX) and the later lateral selective wet oxidation (LSWOX). The process does not oxidize InGaAs or InP to any great extent and the oxidation can be controlled laterally via the use of patterned silica mask layers as the reactants do not diffuse through silica at a significant rate.

The objectives of this investigation are to:

- Design and fabricate PSWOX QC laser arrays with an average output power of at least 100mW at temperatures greater than 77K.
- Compare the optical and electrical performance of PSWOX QC array lasers with that of QC lasers fabricated by wet etching an array of ridge waveguides.

[†] QCLs based on valence band transitions are theoretically possible.

[1] Jérôme Faist, Federico Capasso, Deborah L. Sivco, Carlo Sirtori, Albert L. Hutchinson and Alfred Y. Cho. Quantum Cascade Laser. Science. **264**, 553 (1994).

[2] R. F. Kazarinov and R.A. Suris. Sov. Phys. Semicond. **5**, 207 (1971).

[3] S. J. Caracci, M. R. Krames, N. Holonyak Jr., M. J. Ludowise and A. Fischer-Colbrie. Long wavelength (λ~1.5μm) native-oxide-defined InAlAs-InP-InGaAsP quantum well heterostructure laser diodes. J. Appl. Phys. **75**, 2706 (1994).

[4] C. D. Farmer, P. T. Keightley, C. N. Ironside, C. R. Stanley, L. R. Wilson and J. W. Cockburn. A quantum cascade laser fabricated using planar native-oxide layers. Appl. Phys. Lett. **77**, 25 (2000).

2 Fabrication

2.1 Wafer structure

The gain region of a QCL must be grown by molecular beam epitaxy (MBE), as this is the only process capable of producing the required ~1nm layers and abrupt interfaces. The MBE facilities at Glasgow were under maintenance for the duration of the contract, so wafers grown at the Engineering and Physical Sciences Research Council III-V Semiconductor Central Facility, Sheffield were used.

The growth sequence of wafer M2222, which is designed to lase at ~5 μm, is given in appendix 1. The wafer was analysed by double crystal X-ray diffraction at Sheffield and it was found that the active region periodicity was 46.7 nm, which is within 1% of the intended value of 46.1 nm. Like wafer A1376, which was used in the previous contract, the gain region of this wafer was taken from a design published by the Lucent group[5]. The waveguide layers and contact layers were designed at Glasgow. The differences to wafer A1376 are:

- The tolerance on the substrate doping level has been reduced so that the waveguide losses should be smaller.
- A layer of InP with the correct doping level was grown on top of the substrate prior to the growth of the rest of the layers.
- Sacrificial layers have been introduced above the heavily doped InGaAs layer used to form the top contact, to protect it from the dry etching used during the fabrication of both the wet-etched and PSWOX devices. These layers are designed to be etched away prior to the formation of the contact.
- This InGaAs contact layer is now delta doped rather than bulk doped. This is so that the average doping level can be increased to 10^{20} cm^{-3}, to reduce the contact resistance.

The wafer M1999, which was used for preliminary oxidation tests, was also based on the design of Faist *et al.*[5], but unlike M2222 it did not have any sacrificial layers or InP epitaxial layer. The top contact was a 25 nm InGaAs layer bulk doped with Si to 5×10^{19} cm^{-3}. There were problems during the growth of this wafer that lead to the formation of a high density of surface defects and so it was only of use for testing the oxidation process.

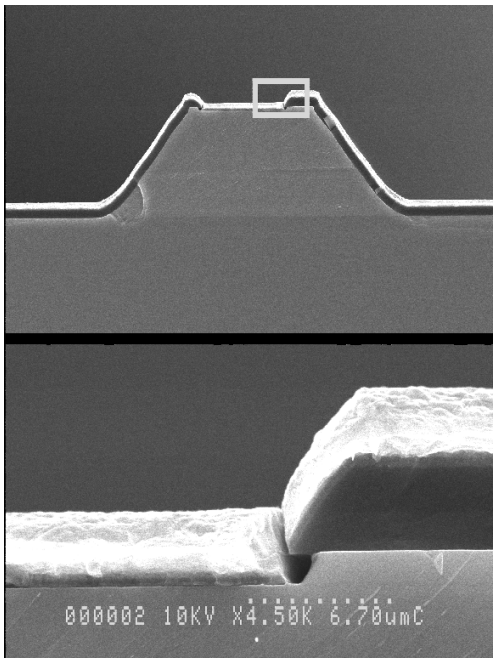
2.2 Device Fabrication

Mesa waveguides were fabricated using the wet-etching procedure described in appendix 2. Results for these devices are given in §3.1 below. An attempt to fabricate devices using the PSWOX process, which is described in appendix 3, was also made. The results of this work is given in §3.2.

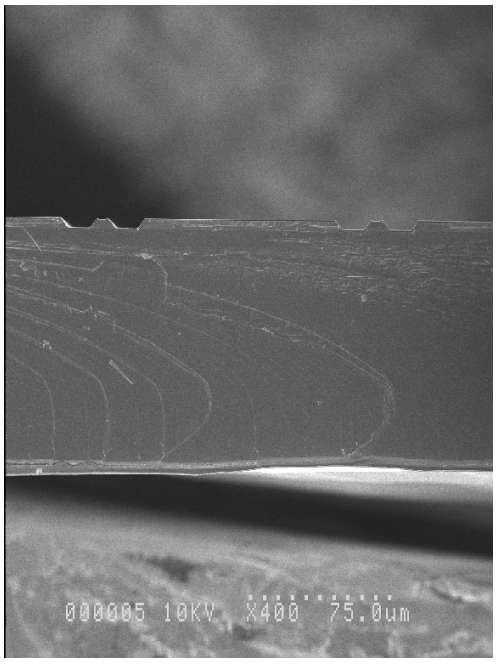
[5] Jérôme Faist, Federico Capasso, Carlo Sirtori, Deborah L. Sivco, James N. Baillargeon, Albert L. Hutchinson, Sung-Nee G. Chu and Alfred Y. Cho. High power mid-infrared (λ~5 μm) quantum cascade lasers operating above room temperature. Appl. Phys. Lett. **68**, 3680 (1996).

3 Results

3.1 Wet-etched mesa waveguides



(a) One wet-etched waveguide. The scale bar only applies to the top half of the image. The lower half is a ten-times enlargement of the indicated area.



(b) A pair of waveguides.

Figure 1. SEM images of a wet-etched QC laser array fabricated from wafer M2222.

A cleaved cross-section through one of the wet-etched waveguides is shown in Figure 1. The moats on either side of the ridge extend all the way down to the InP substrate and a window has been etched all the way through the silica, as desired. The metallization (white) may not have bridged the step properly where the contact window has been etched through the silica (black). This could cause a problem if the devices are mounted using wire bonds, but is less of a problem if the devices are mounted epi-down, with the ridges bonded directly to a heatsink. The cleaving has not produced a completely planar facet, which will increase the mirror loses and hence the threshold current. Fortunately, the defect is at the edge of the waveguide where the light intensity will be lowest. All the waveguides showed this defect for this particular cleave, but the supplied devices were cleaved from a separate part of the wafer.

Using the thickness of the core as a scale bar for the vertical direction shows that the sacrificial layers have been etched to a depth of 129±1 nm, which suggests that the InAlAs-specific etch did not work properly or that the InGaAs-specific etch did not remove all the 130 nm InGaAs layer. Further evidence for this is that if the etches had worked as intended, there would be an undercut under the InGaAs sacrificial layer. The unetched 20 nm InAlAs layer is undoped and will have to breakdown if current to flow through the device.

A 1.316±0.002 mm long 3 ridge wet-etched array was mounted onto a Cu block using In. The electrical connection to the top contact was made by ultrasonically bonding three Au wires to the top of the chip. The sample was then cooled to 90.4 K in a closed-cycle He cryostat and driven with 100 ns long square pulses at a rate of 500 Hz. The light exiting the cryostat through a ZnSe window was detected by a liquid nitrogen cooled HgMnTe detector. At a peak current of 1.6 A, as sensed by a current probe, the detector started to produce a signal. This is an average current density of

3.4 kA cm^{-2} in the central period of the core. Faist *et al.*[6] found a threshold current density of about 1 kA cm^{-2} at 90 K for 3 mm single cavity devices with 8-14 μm wide ridges and the same active region.

The polarisation of the emitted light was investigated by placing a KRS-5 wire-grid polarizer between the detector and the cryostat window. At 77 K and when driven with 100 ns long 4 A pulses at 500 Hz, the greatest intensity was detected when the polarizer was oriented to transmit light with the electric field parallel to the layers in the active region. The selection rules for intersubband optical emission[7], whilst not rigorously obeyed for a non-parabolic band, imply that light emitted by an intersubband device should be strongly polarised perpendicular to the layers. As the polarisation was not in the expected plane and was only weak, it seems likely that the sample was not lasing and was just emitting light by joule heating. The light must have been weakly polarised by reflection from the walls of the cryostat.

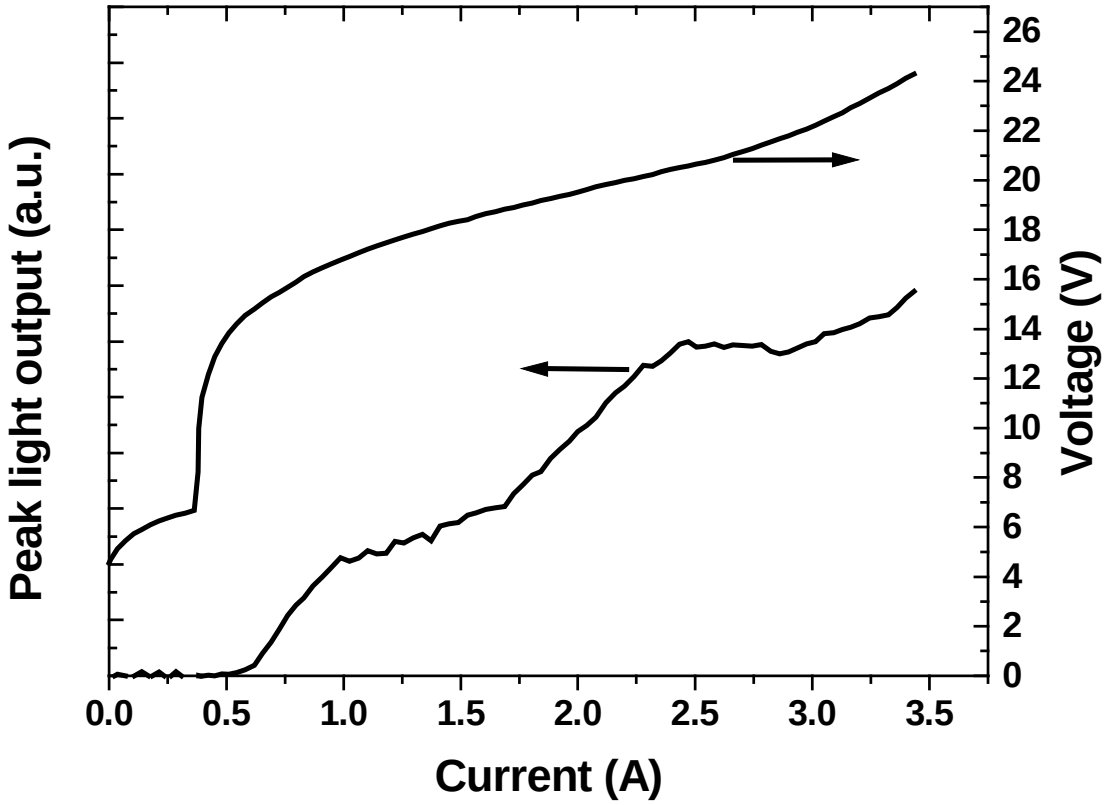


Figure 2. Measurement performed by Damian Carder at Sheffield University Physics department on a single waveguide device fabricated by Corrie Farmer at Glasgow University from wafer M2222. The device was a 2 mm long 20 μm wide wet-etched mesa. The device was driven with 100 ns long square pulses at 3.5 kHz.

Some measurements performed by Sheffield University on another device fabricated from wafer M2222 are given in Figure 2. The sudden increase in the resistance of this device at around 7 V should not be present – there must be a problem the wafer or fabrication. Spectral measurements on this device performed at Sheffield using a step-scan Fourier transform infrared spectrometer did not detect the intense, narrow spectral line characteristic of lasing. The light measured in Figure 2 was attributed to joule heating.

The failure of both of these and other devices to lase strongly suggests that wafer M2222 has not been grown properly.

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[6] Jérôme Faist, Federico Capasso, Carlo Sirtori, Deborah L. Sivco, James N. Baillargeon, Albert L. Hutchinson, Sung-Nee G. Chu and Alfred Y. Cho. High power mid-infrared (λ~5 μm) quantum cascade lasers operating above room temperature. Appl. Phys. Lett. **68**, 3680 (1996).
[7] John H. Davies. The Physics of Low-Dimensional Semiconductors. Cambridge University Press. (1999).

3.2 PSWOX waveguides

The first attempt at producing PSWOX waveguides was made with wafer M1999. A different procedure to that detailed in appendix 3 was used to form the structure shown in Figure 3. Instead of using the mask shown in Figure 5, a mask that just formed an array of narrow bars of silica was used. It was hoped that the silica could have been removed using dry etching and a contact deposited directly onto the surface, omitting the second photolithography step. As can be seen in Figure 3, the InAlAs cladding was converted to native oxide to a reasonable depth. Unfortunately, however, an unidentified material was deposited on top of the silica mask (the silica is the darkest layer in Figure 3). This material proved to be impervious to dry etching with C_2F_6 plasma and to various wet-etches, so it was not possible to make an electrical contact to the top of the waveguides.

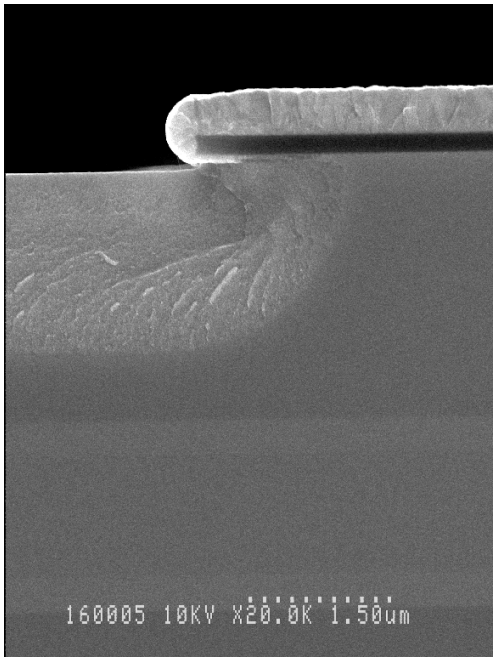
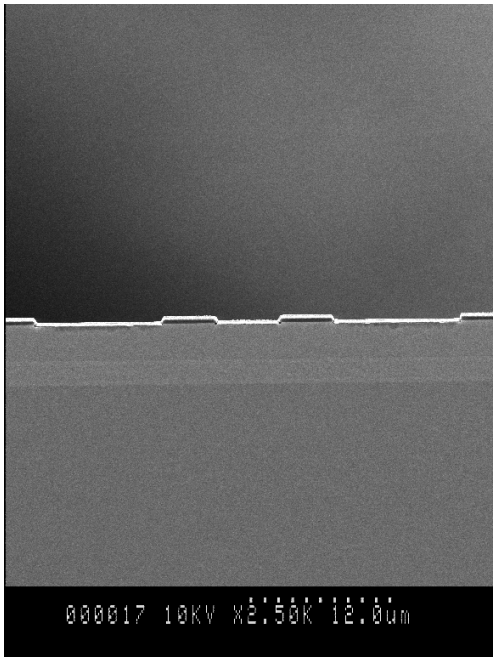


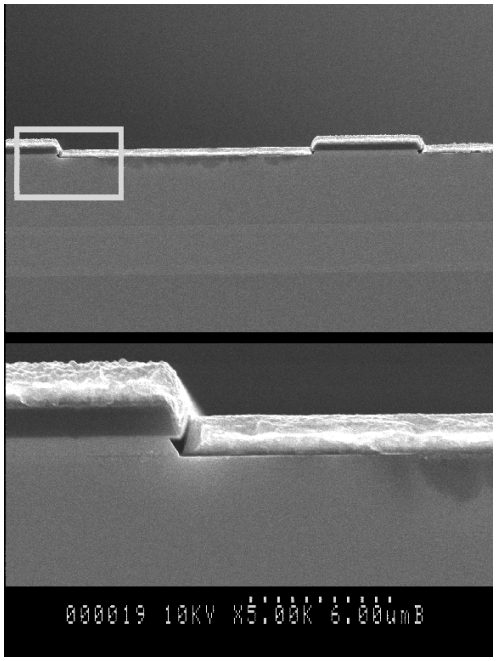
Figure 3. PSWOX device fabricated from wafer M1999. The sample was oxidized for 1451 min with a N_2 flow rate of 80 standard cubic centimetres per minute.

To try and circumvent this problem, the amount of material converted to the native oxide was reduced. This was done by using the mask shown in Figure 5 to keep the material between the waveguides covered with silica during the oxidation. Wafer M2222 was used, along with the procedures listed in appendix 3, the results being shown in Figure 4. This time the oxidation is much shallower and the interface between the native oxide and the InAlAs is far from planar. Closer inspection of the region shown in the lower half of Figure 4(c) showed that the n^+ InGaAs cap and digital grading from InGaAs to InAlAs had not been completely removed. Since InGaAs acts as a barrier to wet oxidation, this is the probable explanation of the poor quality of the oxide. As the InAlAs has not been oxidized properly, it is not possible to say if increasing the fractional area masked off by silica has prevented the formation of the unidentified layer in Figure 3.

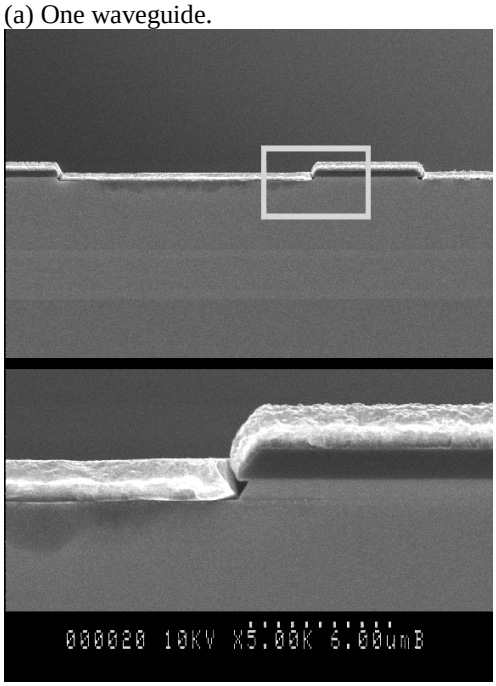
As the native oxide is not providing electrical or optical confinement, no devices were tested.



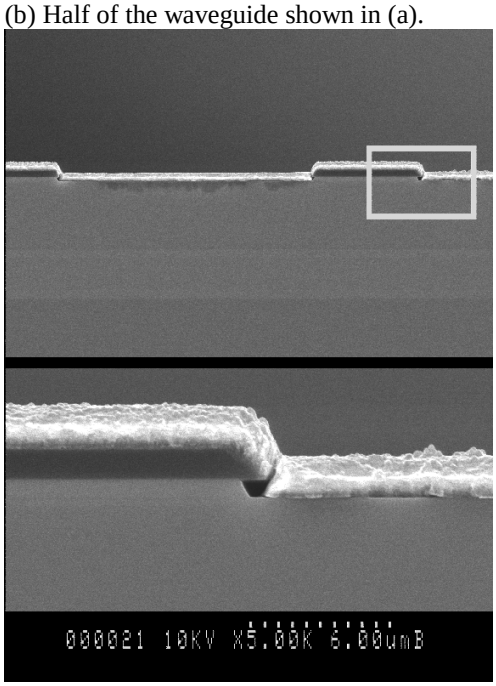
(a) One waveguide.



(b) Half of the waveguide shown in (a).



(c) Half of the waveguide shown in (a).



(d) Half of the waveguide shown in (a).

Figure 4. PSWOX device fabricated from wafer M2222. The sample was oxidized for 1454 min with a N_2 flow rate of 250 standard cubic centimetres per minute.

4 Conclusions

As no working devices were produced, it was not possible to achieve the aims given in §1. It was not possible to produce a significant amount of IR radiation at 77 K. Neither was it possible to compare the results of the wet-etching and PSWOX waveguide formation technologies. It was not possible to reproduce the results obtained by Farmer *et al*.[8] in the time available as a number of problems were found:

- A substance was deposited on top of the silica oxidation mask, that prevented its removal by dry etching.
- It was found that the native oxide was attacked by the etchant used to selectively etch the InAlAs sacrificial layers in wafer M2222, so the process had to be repeated in a modified form to allow for this.
- The cap layers of wafer M2222 were not etched away properly, which prevented the formation of the native oxide.

Further development of the PSWOX process is required if it is to be used to reliably fabricate QC laser arrays, however it is not clear that this is worth the trouble, since it is not possible to oxidise the Ga-rich core region, and so it is not possible to confine the current as it passes through the core. The optical losses of the native oxide have also not been characterised.

Finally, even if the processes had been carried out successfully, the devices probably would not have lased, as wafer M2222 has not been fabricated into any working devices by the author or by other members of the quantum cascade laser group at Glasgow.

[8] C. D. Farmer, P. T. Keightley, C. N. Ironside, C. R. Stanley, L. R. Wilson and J. W. Cockburn. A quantum cascade laser fabricated using planar native-oxide layers. Appl. Phys. Lett. **77**, 25 (2000).

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Appendix 1 – Wafer M2222 MBE growth sequence

All doped layers were doped with Si, except for the substrate, which is Sn doped. All layers were grown lattice-matched to the InP substrate. $In_{0.53}Ga_{0.47}As$ and $In_{0.52}Al_{0.48}As$ have been shortened to InGaAs and InAlAs in this table and the rest of this report.

	repeats	thickness/Å	T_S/°C	[dopant]/cm^{-3}
InGaAs	1	1300	500	
InAlAs	1	200	500	
InGaAs	1	20	500	
2×10^{13} cm^{-2} δ-doped layer	5	3	500	
InGaAs		17	500	
InGaAs	1	50	500	7×10^{18}
InGaAs	1	42	500	7×10^{18}
InAlAs	1	9	500	7×10^{18}
InGaAs	1	36	500	7×10^{18}
InAlAs	1	15	500	7×10^{18}
InGaAs	1	30	500	7×10^{18}
InAlAs	1	21	500	7×10^{18}
InGaAs	1	21	500	7×10^{18}
InAlAs	1	30	500	7×10^{18}
InGaAs	1	12	500	7×10^{18}
InAlAs	1	39	500	7×10^{18}
InGaAs	1	6	500	7×10^{18}
InAlAs	1	45	500	7×10^{18}
InAlAs	1	12000	500	7×10^{18}
InAlAs	1	7000	500	3×10^{17}
InAlAs	1	6000	500	2×10^{17}
InAlAs	1	45	500	2×10^{17}
InGaAs	1	6	500	2×10^{17}
InAlAs	1	39	500	2×10^{17}
InGaAs	1	12	500	2×10^{17}
InAlAs	1	30	500	2×10^{17}
InGaAs	1	21	500	2×10^{17}
InAlAs	1	21	500	2×10^{17}
InGaAs	1	30	500	2×10^{17}
InAlAs	1	15	500	2×10^{17}
InGaAs	1	36	500	2×10^{17}
InAlAs	1	9	500	2×10^{17}
InGaAs	1	42	500	2×10^{17}
InGaAs	1	3000	500	1×10^{17}
InAlAs	25	15	500	
InGaAs		47	500	
InAlAs		30	500	

InGaAs		40	500	
InAlAs		30	500	
InGaAs		23	500	
InAlAs		23	500	
InGaAs		22	500	
InAlAs		22	500	2×10^{17}
InGaAs		20	500	2×10^{17}
InAlAs		20	500	2×10^{17}
InGaAs		20	500	
InAlAs		23	500	
InGaAs		19	500	
InAlAs		28	500	
InGaAs		19	500	
InAlAs		50	500	
InGaAs		10	500	
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InGaAs	1	47	500	
InAlAs	1	22	500	
InGaAs	1	40	500	
InAlAs	1	30	500	
InGaAs	1	23	500	
InAlAs	1	23	500	
InGaAs	1	22	500	
InAlAs	1	22	500	2×10^{17}
InGaAs	1	20	500	2×10^{17}
InAlAs	1	20	500	2×10^{17}
InGaAs	1	3000	500	1×10^{17}
InAlAs	1	3	500	2×10^{17}
InGaAs	1	48	500	2×10^{17}
InAlAs	1	6	500	2×10^{17}
InGaAs	1	45	500	2×10^{17}
InAlAs	1	9	500	2×10^{17}
InGaAs	1	42	500	2×10^{17}
InAlAs	1	12	500	2×10^{17}
InGaAs	1	39	500	2×10^{17}
InAlAs	1	15	500	2×10^{17}
InGaAs	1	36	500	2×10^{17}
InAlAs	1	18	500	2×10^{17}
InGaAs	1	33	503	2×10^{17}
InP	1	1000	513	1×10^{17}
InP substrate	-	3500000	-	1×10^{17}

Appendix 2 - Fabrication procedure for wet-etched mesa waveguides

1. Scribe, cleave and clean wafer.
2. Spin on photoresist.
3. Expose using the mask plate shown in Figure 5 and develop. The mask plate used had D_{array} = 150 μm.
4. Hard bake the resist to improve its resistance to acids.
5. Form the ridges by etching down to the InP layer using 1:8:1 H_2O: 30% hydrogen peroxide solution: 85% orthophosphoric acid.
6. Remove the photoresist in acetone.
7. Deposit 400 nm of silica by plasma-enhanced chemical vapour deposition.
8. Spin on photoresist.
9. Open windows in the photoresist on top of the wet-etched ridges.
10. Hard bake the photoresist.
11. Dry etch the contact windows through the silica using C_2F_6 plasma.
12. Remove photoresist with acetone.
13. Etch away the sacrificial layers using the following selective wet etches:

    a. 1:1 H_2O : 37% hydrochloric acid to deoxidise surface.
    b. 1:1 30% hydrogen peroxide solution : citric acid solution (made up with equal masses of citric acid powder and water) to remove the InGaAs layer.
    c. 1:1 H_2O : 37% hydrochloric acid to remove the InAlAs layer.

14. Dry in N_2 purged oven at 90 °C.
15. Clean the surface using Ar plasma and then, without exposing to air, deposit 30 nm Ti/ 33 nm Pd/ 20 nm Ti/ 240 nm Au by e-beam evaporation.
16. Thin substrate down to 200 μm using 9 μm alumina powder suspended in water.
17. Thin substrate down to 150 μm using 3 μm alumina powder suspended in water.
18. Clean sample.
19. Protect the top contact with photoresist and etch the back of the substrate in 10:1:1 H_2O : 47% hydrobromic acid : 37% hydrochloric acid.
20. Clean surface using Ar plasma and then, without exposing to air, sputter on a 30 nm Ti/60 nm Pt/250 nm Au contact.

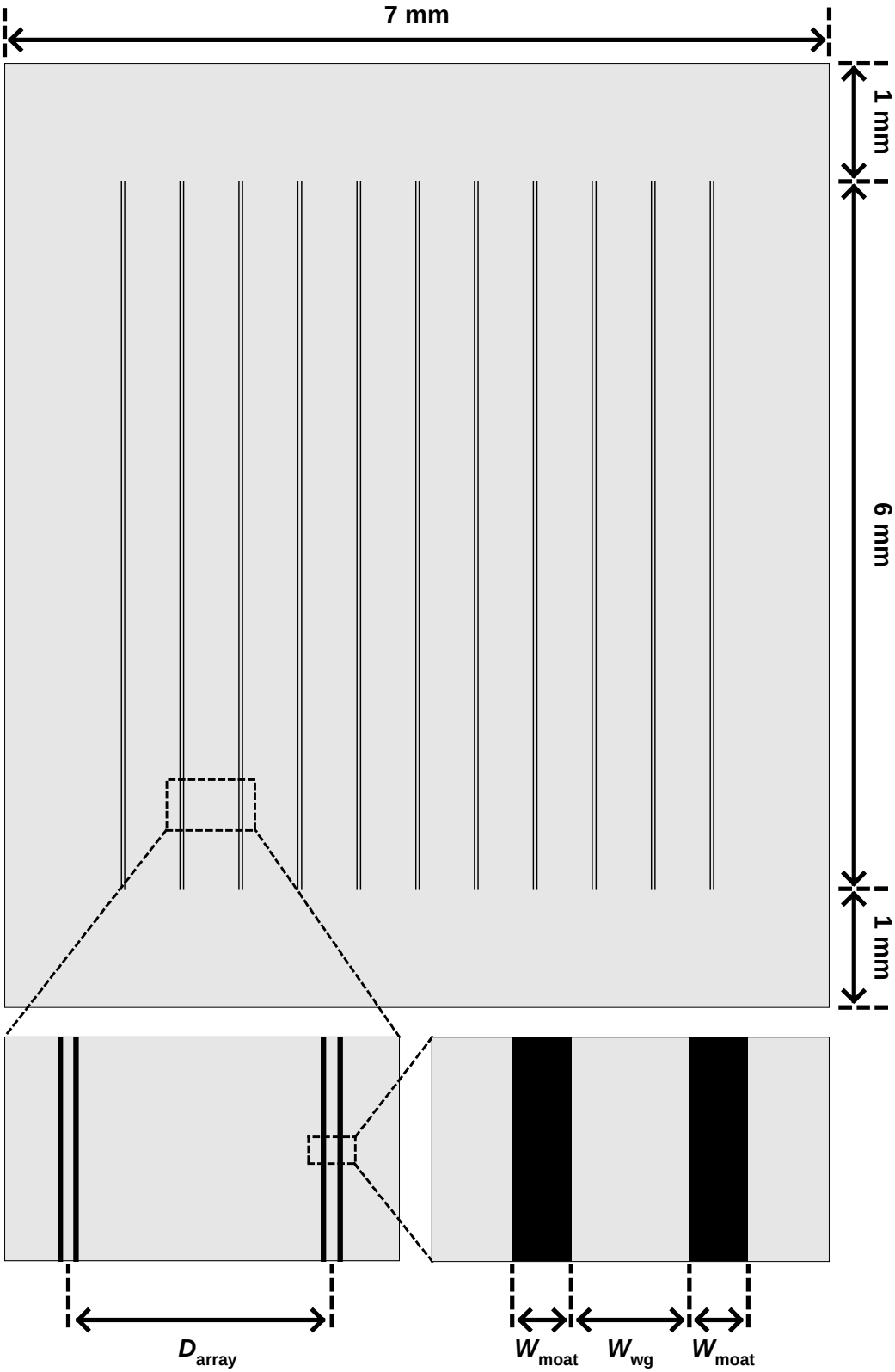

Figure 5. Schematic diagram of mask plate used to form waveguides for both the wet etching and PSWOX processes. The grey regions are opaque and the black regions are transparent.

Appendix 3 - Fabrication procedure for native oxide waveguides

1. Scribe, cleave and clean the wafer.
2. Deposit 200 nm of silica by plasma-enhanced chemical vapour deposition.
3. Spin on photoresist.
4. Expose the resist using the mask plate shown in Figure 5 above. The mask plate used had $D_{array} = 150\ \mu m$.
5. Develop photoresist.
6. Hard bake photoresist to increase its resistance to the silica etch.
7. Etch through the silica with 4:1 buffered silica etch to form a mask for the wet oxidation.
8. Strip the photoresist with acetone.

Since the layers used to form the gain region in a QC laser are only a few atomic mono-layers thick, interdiffusion of the InGaAs and InAlAs layers could be a problem during the approximately day-long period that the device is held at an elevated temperature. The furnace is set to 500 °C during the oxidation and the steam entering the furnace must be above atmospheric pressure and so must be above 100 °C, so the gain region is somewhere between 100 °C and 500 °C. During the MBE growth of the wafer, which occurs over a similar timescale, the *surface* of the sample is held at 500 °C (see appendix 1). As the temperature of the sample is lower during the PSWOX process than during the MBE growth, it seems reasonable to expect the gain region is not too badly affected by the PSWOX process. Further evidence that the gain region is not seriously affected by the PSWOX process is given by the successful fabrication of QC lasers using native oxide by Farmer *et al.* [9].

9. Immediately prior to oxidation, remove any sacrificial layers, the InGaAs contact layer and the digital grading to InAlAs using the following wet etches:

    a. 200:1:1 H2O: 30% hydrogen peroxide solution: 98% sulphuric acid.
    b. 98% sulphuric acid.

    Rinse in water and blow dry after both etches.

10. Oxidize using the set-up illustrated in Figure 6. The heated silica glass tube reduces the amount of condensation reaching the furnace.

    a. Heat fresh reverse osmosis purified water to a few degrees below its boiling point with zero grade N_2 bubbling through it.
    b. Load sample into furnace as soon as possible after etching the cap layer and pass N_2 over it.
    c. Wait until the furnace has been purged of all air and then set the sample heater to 500 °C.
    d. Wait 10 min to allow the walls of the furnace to heat up.
    e. Change valves to pass N_2 through bubbler then over sample.
    f. Oxidize for the required length of time.
    g. Change valves to pass dry N_2 over sample.
    h. Wait until any water that has condensed in furnace has evaporated.
    i. Switch off furnace.
    j. Unload sample when cool.

[9] C. D. Farmer, P. T. Keightley, C. N. Ironside, C. R. Stanley, L. R. Wilson and J. W. Cockburn. A quantum cascade laser fabricated using planar native-oxide layers. Appl. Phys. Lett. **77**, 25 (2000).

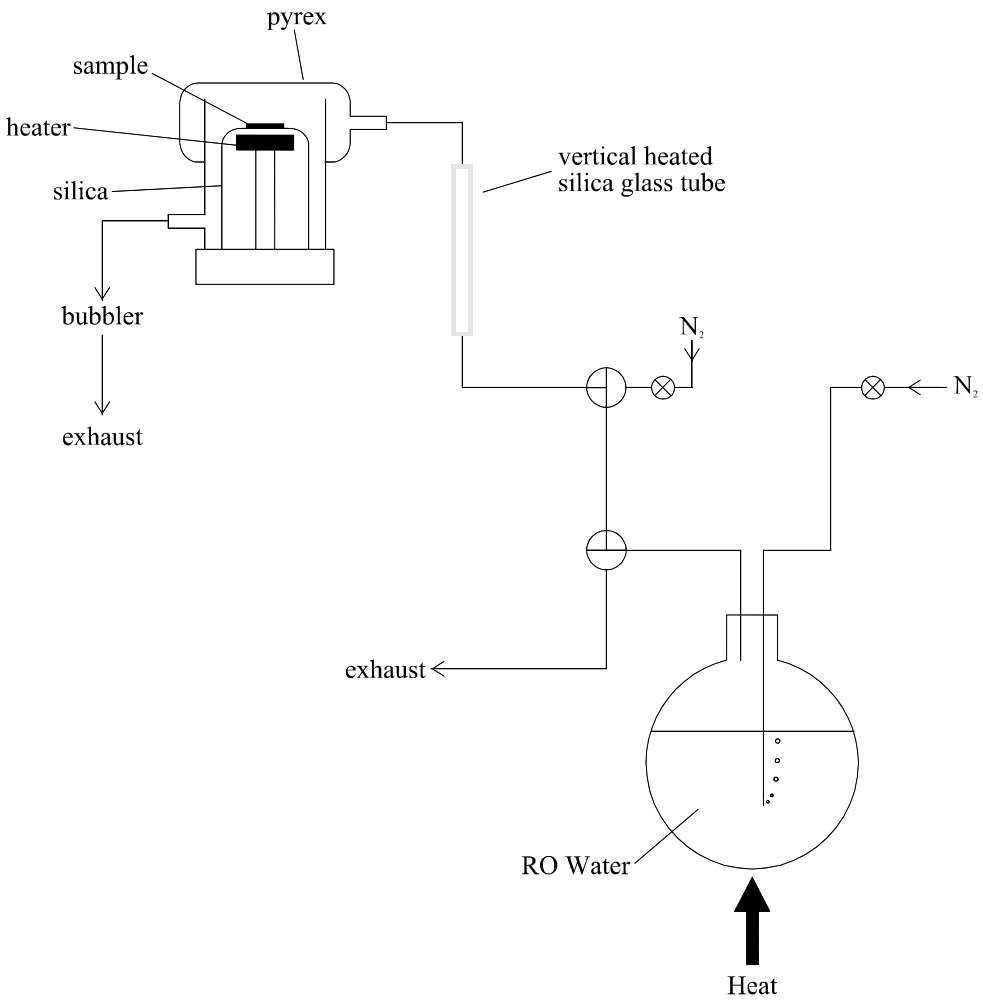

Figure 6. Oxidation apparatus. N.B. it is possible to squirt boiling water out of this apparatus if the valves are incorrectly operated and the tubing supplying N_2 to the heated flask is blown off by pressure applied by the line supplying dry N_2 to the furnace. It would also be possible to implode any glassware by cooling the flask with all the valves closed.

11. Spin on photoresist.
12. Open windows just narrower than the central silica bars.
13. Dry etch a contact window through the silica using C_2F_6 plasma.
14. Etch away the damage done to the sacrificial layers by the dry etching using the same procedure used for the wet-etched process.
15. Strip photoresist with acetone.
16. Dry in N_2 purged oven at 90°C.
17. Clean surface using Ar plasma and then, without exposing to air, deposit 30 nm Ti/ 33 nm Pd/ 20 nm Ti/ 240 nm Au by e-beam evaporation.
18. Thin substrate and apply back contact.

Appendix 5 - Description of supplied devices

Two wet-etched arrays of QC lasers have been supplied. One array consists of three stripes and the other of five.

- Both devices are the same length, which is nominally 2 mm.
- Both devices are 150±20 μm thick.
- The waveguides are separated by 150 μm
- Remember not to damage the facets when picking up the devices.
- The sheet resistance of the top contact is 0.23 $\Omega\,\square^{-1}$ ±40%.
- The back contact is biased positive with respect to the top contact when the device is forward biased.
- Any resistors used in the output stages of the driving circuitry must be non-inductive – the voltage spikes created by wire-wound resistors during pulsed operation will destroy the device.
- The wafer the devices are fabricated from is probably incapable of lasing.
- The undoped 20 nm sacrificial InAlAs layer near the surface of the wafer was not removed during the fabrication and will have to breakdown if the devices are to pass any current.